



AFRL-OSR-VA-TR-2015-0086

Graphene Synthesis and Characterization 130060

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04/08/2015
Final Report

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REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188	
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1. REPORT DATE (DD-MM-YYYY) 03-04-2015		2. REPORT TYPE Final Report			3. DATES COVERED (From - To) Mar, 01, 2014-Mar, 01, 2015	
4. TITLE AND SUBTITLE Grant: Graphene synthesis and caracterización				5a. CONTRACT NUMBER		
				5b. GRANT NUMBER FA9550-13-1-0060		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Andrea Cortés				5d. PROJECT NUMBER		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Universidad Técnica Federico Santa Maria (UTFSM) Avenida España 1680, Valparaíso, Chile					8. PERFORMING ORGANIZATION REPORT NUMBER DUNS 1435774726	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) AF Oficce Scientific Research 875 N. Randolph St. Room 3112 Arlington VA 22203					10. SPONSOR/MONITOR'S ACRONYM(S) AFOSR	
					11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT DISTRIBUTION A Limited Distribution						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT <p>Here is reported graphene synthesis by two methods, Chemical Vapor Deposition (CVD) using Acetylene as a Carbon source onto copper substrates, and the exfoliation of graphite (chemical and electrochemical). These methods have a great importance for applications in devices, because can be properly scaled to industrial level. It was studied the experimental parameters involved in the growth of graphene. By Raman spectroscopy was made the characterization of material obtained. CVD method allow to grow single- and few-layer graphene on copper. On the other hand, the exfoliation method produced multi-layer graphene flackes.</p>						
15. SUBJECT TERMS Graphene, CVD, Copper, Acetylene, Exfoliation, Tranfer						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES 17	19a. NAME OF RESPONSIBLE PERSON Andrea Cortes	
a. REPORT C	b. ABSTRACT C	c. THIS PAGE C			19b. TELEPHONE NUMBER (Include area code) +56 32 2654628	

AFOSR Final Performance Report

To: technicalreports@afosr.af.mil

Subject: Final Performance Report to Dr. Brett Pokines

Grant Title: Graphene synthesis and characterization

Award Number: FA9550-13-1-0060

Reporting Period: Mar. 01, 2014 to Mar. 01, 2015

Gov. Program Manager: Dr. Brett Pokines

Principal Investigator: Dr. Andrea Cortés, Physics Department, UTFSM,
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Recipient Organization: Universidad Técnica Federico Santa María, Avenida España 1680,
Valparaíso, Chile.

DUNS: 143574726

Annual accomplishments: see below

Archival publication during reporting period: see below

Changes in research objectives: None

Include any new discoveries, inventions, or patent disclosures during this reporting period: None.

PROPOSAL:

GRAPHENE SYNTHESIS AND CHARACTERIZATION

1. ABSTRACT

The challenges of graphene synthesis for application at industrial level are closely related to the synthesis of good quality material (good electrical properties and stability) and the ability to transfer it onto large areas without changing their physical properties. We study two methods of synthesis, which can be properly scaled to industrial level, such as chemical vapor deposition (CVD) and chemical exfoliation of graphite. Also, we explore graphene transfer onto dielectric substrates. This point turns out to be a key step in all applications.

2. INTRODUCTION

Graphene is a material formed by a single atomic layer of carbon atoms arranged in a hexagonal structure (sp^2 hybridized carbon). The mechanical and chemical stability that graphene has, together with its electrical and optical properties, makes it an interesting material from the point of view of technical applications [1]. For the synthesis of graphene several methods have been developed, including micro-mechanical exfoliation of highly oriented graphite (HOPG), chemical exfoliation of graphite, thermal decomposition of SiC and CVD over metallic substrates [1-3].

2.1 CVD graphene synthesis

The CVD synthesis is both simple and inexpensive. It allows the production of graphene over large areas. It involves the decomposition of gaseous hydrocarbons (CH_4 , C_2H_2 , CH_3OH , among others) at elevated temperature. The metallic substrate acts as a catalyst for the decomposition reaction leaving carbon on the surface. However, it has been difficult to control the number of graphene layers produced. In recent years there has been extensive use of Cu as catalyst, because it provides a better control of the number of layers of graphene, which is directly related to the low solubility of carbon in copper [4,5].

Along with the growth of graphene by CVD, a methodology which allows the transfer from the growth substrate to other substrate of interest has been developed (such as dielectric substrates). It includes the use of a protective polymer such as polymethylmethacrylate (PMMA) [6,7]. The process involves applying the polymer on the surface of graphene which remains bound to the metal, once cured the polymer, the metal is chemically dissolved and graphene remains on the polymer. Finally, the graphene-polymer composite is placed on a substrate of interest on the graphene side and the polymer is dissolved with an organic solvent.

This is a topic of continuous research in recent years. Sukang Bae et al. [8] reported a method of producing "roll-to-roll" with which they obtained graphene films (monolayers predominantly) over areas as large as 30 inches. Graphene was grown by CVD on copper, then transferred to a thermal protective film passing between two rollers and then copper is removed by chemical attack. The authors' goal was making graphene transparent electrodes.

Gang Hee Han et al. [9] used the "roll-to-roll" transfer system in a single step, using the properties of the thermoplastic polymer. These authors were able to grow a graphene sheet (mixed mono-and bilayers, where the formation of monolayers was dominant) over an area as large as 75x85 cm².

2.2 Exfoliation of graphite

An alternative method for preparing industrial scale graphene is chemical exfoliation of graphite [1,3]. Exfoliating graphite in aqueous solutions is difficult due to their hydrophobic character; however, when subjected to a chemical oxidation, it is possible to separate the layers of graphite oxide in water by extended sonication. The final product is graphene oxide suspended in water. The disadvantage of graphene oxide is its difficulty to incorporate it into organic polymers to form composites. This material is also an electrical insulator. Therefore, for applications in devices, the graphene oxide must be reduced, which presents a coagulation problem [10].

S. Stankovich et al. [11] reported the chemical modification of graphite oxide by treatment with isocyanate, which reduced the hydrophilic character of the graphene sheets due to the formation of various functional groups. As a result of the chemical modification oxides do not exfoliate in water but form stable dispersions in polar aprotic solvents. The graphene sheets obtained by this method are wrinkled and at times bent compared to graphene sheets prepared on metal substrates by CVD [11].

Another method of interest is that proposed by Ching-Yuan Su et al. [12] and involves the electrochemical exfoliation of graphite foil to obtain high quality graphene. The lateral size of the sheets is as large as 40 microns (which reduces the number of joints between sheets). The number of layers of graphene sheets varies between 1 and 4, being more frequently observed bilayer graphene (65%). These authors reported that graphene obtained by this method has small amounts of oxygen species, which can be reduced in hydrogen at 450°C.

Using the above techniques (CVD and chemical/electrochemical exfoliation) our goal is to study the parameters involved in the synthesis of graphene and propose a suitable methodology to prepare this material over large areas with future projections for the design of devices, whether optical, electronic, etc.

3. EXPERIMENTAL

3.1- Synthesis of graphene by the CVD technique

Copper foil (25 microns thick and 99.98% purity) and thin copper films (by sputtering) were used as substrate without any treatment cleaning. The substrates were put inner a quartz tube of three zones furnace and was programmed at 600-1000°C with a heating rate of 90°C/min. Acetylene gas was used as a carbon source with argon and hydrogen at 1 mbar of pressure. We studied the parameters involved in the process such as: temperature, gas flow, synthesis time.

3.2- Transfer the graphene from the copper to other substrates (dielectric)

After synthesis, graphene kept on copper substrate and a thin polymer film was formed onto its surface by spin-coating technique. The polymer used was PMMA and was prepared by dissolving 60 mg of Poly(methyl methacrylate) in 1 ml of chlorobenzene. The polymer solution was dropped on graphene-copper at 2400 rpm for 15 seconds and then allowed to cure for 12 hours at ambient room temperature. Then the graphene was detached from copper by electrochemically and the PMMA was dissolved with acetone.

3.3- Exfoliation of graphite (chemical or electrochemical)

3.3.1- Chemical:

We used chemical synthesis of graphene by Hummer Modified method. Graphite natural was used as source of graphene. The graphite is put in a solution, compound of sulfuric and nitric acid (concentrate), a solution of K_2MnO_4 (5%) and a hydrazine solution (1%). For each treatment, the temperature varied: 20-120°C). Then, in each procedure flakes were collected by centrifugation, eliminated the supernatant and washed with deionized, this procedure is repeated many times.

3.3.2- Electrochemical:

The electrochemical exfoliation was performed using a system conformed by a power source and electrochemical glass cell. Platinum wire was used as counter-electrode, HOPG as work electrode and a solution of sulfuric acid (5%) as electrolyte. For the exfoliation process was applied a potential pulses.

3.4- Characterization

I was conducted primarily by Raman spectroscopy using a laser of 532 nm Scanning Electron Microscopy (SEM) and Optical microscopy.

4. FUNDING AND RESEARCHERS

4.1. All Funding Sources:

- Synthesis: Air Force Office of Scientific Research, USA: FA9550-13-1-006 (USA) and FONDECYT 11110522 (Chile).
- Characterization: FONDECYT 11110522 (Chile) and FONDEF D11I1213 (Chile)

4.2. Researchers:

- Andrea Cortés
- Carlos Celedón

5. FACILITIES

At the sponsoring institution (Physics department, UTFSM), we count with several equipment to develop this proposal.

- A Chemical Vapor Deposition (CVD) system with digital controllers. This CVD system consists a horizontal quartz tube furnace (three-stage) connected to gas flow lines (argon, hydrogen, acetylene) maximum work temperature is 1100°C.
- A Raman Spectrometer (Renishaw), laser $\lambda=532$ nm.
- A Optical microscope (Leica DM500).
- A spin-coater systems (Laurell, model WS-650MZ-23NPP) for preparing polymer thin film.
- A Power source (home-made) for synthesis electrochemical.
- A Scanning Electron Microscope (SEM) (EVO MA from Carl Zeiss).

6. RESULTS AND DISCUSSION

6.1.- CVD synthesis of graphene

i. Grain size copper

The treatment previous to the graphene synthesis was carried out. The annealing of copper foil improves the nucleation and growth of graphene, which dependent strongly on the annealing time and growth temperature [13]. The annealing process causes an increase in the grain size of the copper foil as received, which can be seen in the optical image (Fig.1). However, we did not achieve a homogeneous grain size. The substrate present a few grains close to 100 micrometers size, while smaller size grains predominate. The shape of the grains is not regular, showing different morphologies.

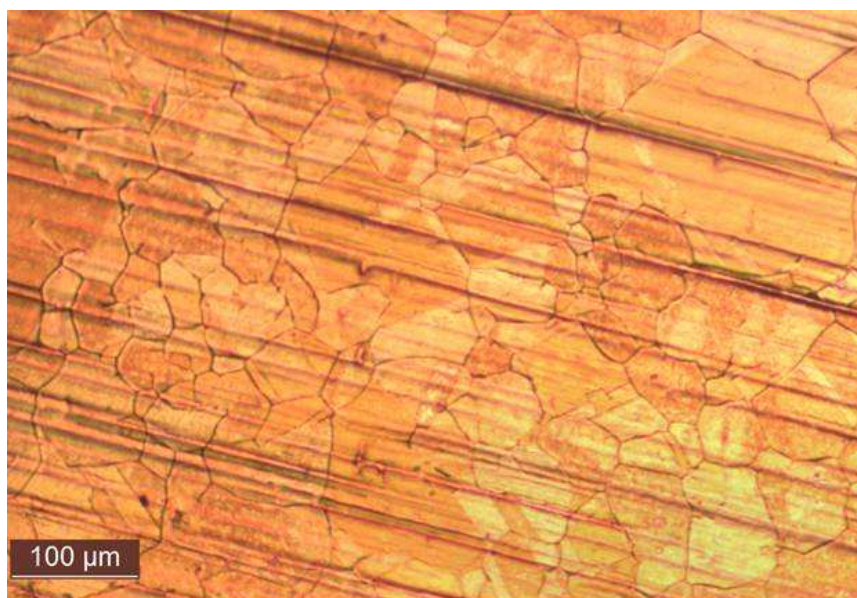


Figure 1. Optical image of a copper foil after thermal treatment at 700°C for 30 minutes in hydrogen flux (100 sccm).

ii. Growth temperature effect

To study the effect of synthesis temperature, we performed the growth at different temperatures in the same condition of synthesis (fluxes and time), see Fig.2. At 600°C is obtained amorphous carbon, distinguishing the D and G bands with the same intensities and the absence of the 2D band, with a poor surface coverage. While at

850°C begins to observe the appearance of 2D band, maintaining a high intensity of D band. A 950°C is obtained graphitic material, distinguishing the three bands; D, G and 2D. At this temperature the D band and G are sharp and they are separated, the ratio of intensities D/G is less than in the samples at low temperature and the spectrum indicate multilayer. A 1000°C is obtained bi-layer graphene, the ratio of D/G is less than at 950°C.

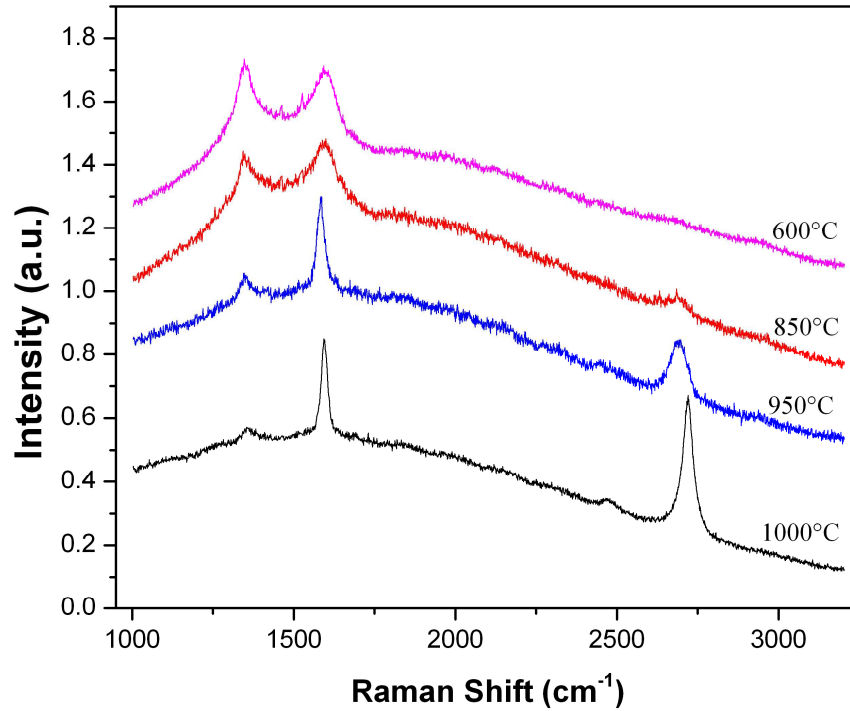


Figure 2. Raman spectra for different temperatures of synthesis. The spectra were taken for graphene on copper ($\lambda=532$ nm).

The G band sharpens with the growth temperature increase, while the ID/IG ratio decreases. The optimum temperature for graphene synthesis is 1000°C, the material has fewer defects (low intensity of the band D) and the presence of bilayer graphene [14].

iii. Effect of Hydrogen flux

In the experiments, during the graphene growth process on copper foil, the acetylene and argon fluxes were stayed constant at 5 and 200 sccm, respectively. The hydrogen flow was varied between 20-120 sccm to study hydrogen effect on growth (TABLE I).

Table 1	
Sample	H ₂ /sccm 20 min
M1_1	80
M1_2	100
M1_3	120

In the experiment set (TABLE 1), hydrogen (60 sccm) and argon (200 sccm) gases were injected into the furnace. Once the system reached 1000 °C, acetylene (5 sccm) was introduced, maintained the argon flow and then, the hydrogen flow was adjusted at 80, 100 and 120 sccm during 20 minutes for the samples M1_1, M1_2 and M1_3, respectively. At last, the acetylene flux was closed, the hydrogen flow was decreased to 20 sccm and the argon flow was maintained in 200 sccm, the system was cooled using this mixture of hydrogen and argon up to room temperature.

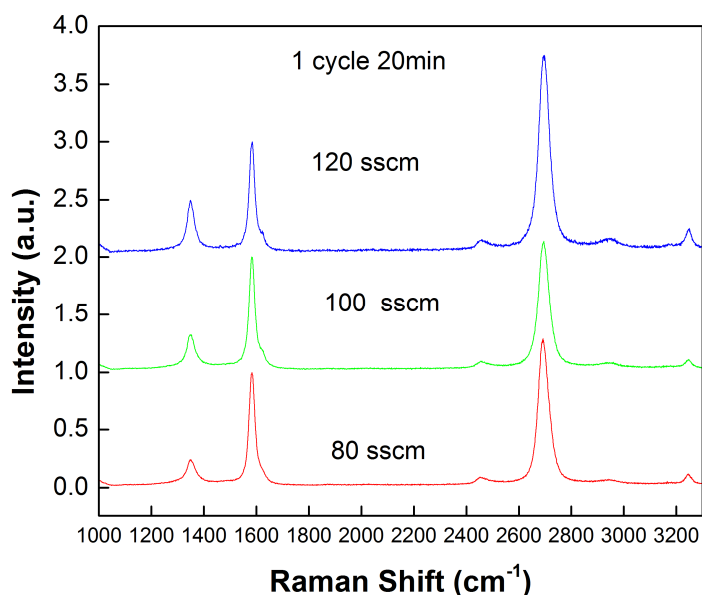


Figure 3.- Raman spectra of graphene films that were transferred from copper foil to SiO₂ (300 nm) substrate. Samples were synthesized by CVD process at different H₂ flow rates from 80, 100 or 120 sccm for 20 min. The flow is maintained during whole synthesis.

From these experiments we can conclude that using a high flow of hydrogen (120 sccm) for a long time (20 minutes), graphene has a high level of defects, the ratio of intensities between D band and G is about 0.5 (Fig 3), the ratio of intensities G/2D is ~0.5 and 2D band is positioned at 2695 cm⁻¹. As the hydrogen flow decreases, is

obtained a material with fewer defects, for a flow of 80 sccm we obtained a lower intensity of D band, ratio D/G intensity band is ~ 0.25 .

iv. Effect of size copper foil

For the synthesis of copper foils large, we using samples of $10 \times 10 \text{ cm}^2$, and observed that the growth is not homogeneous in terms of coverage and in regard to material quality. The center of the sample presents homogeneous growth of bi- and three-layer graphene, but in areas close the sheet edge, material has more defects, together with the presence of multilayers (2-3).

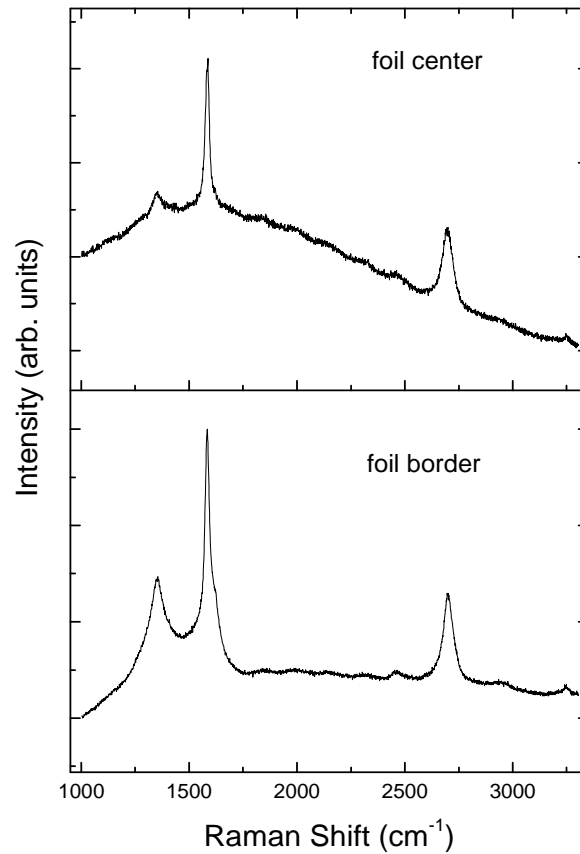


Figure 4.- Raman spectra of graphene on copper foil substrate of $10 \times 10 \text{ cm}^2$. Spectra were measured in various locations on the copper foil and be show the main results ($\lambda = 532 \text{ nm}$).

6.2.- Graphene transfer

To perform the Raman characterization, a transfer process was performed of graphene from the copper substrates to the SiO₂/Si substrates. For this purpose, first, the samples of graphene-copper are covered for PMMA layer (protective polymer) by spin-coating. Then, we used an electrochemical technique, using two electrodes, one of them is the graphene-copper foil (cathode) and the another one is a graphite plate (anode) and both electrodes were submerged in a sodium sulphite solution (5%) as electrolyte and it is applied voltage pulses of 5 V for 20 minutes, until the graphene is detached from the copper and stays floating over the solution. Then the graphene-PMMA is washed with deionized water many times for eliminate the salt from electrolyte (Fig.5).

This process involves the generation of small bubbles of hydrogen which allow the separation of graphene from the metal substrate, as described in Fig. 6. Using this technique, the generation of defects in the material is not observed, because the voltage pulse is applied for a few seconds and the graphene is not affected by electrolyte as in the case of using Fe (III) to dissolve the copper [15]. Therefore this is a simple, clean and fast technique.

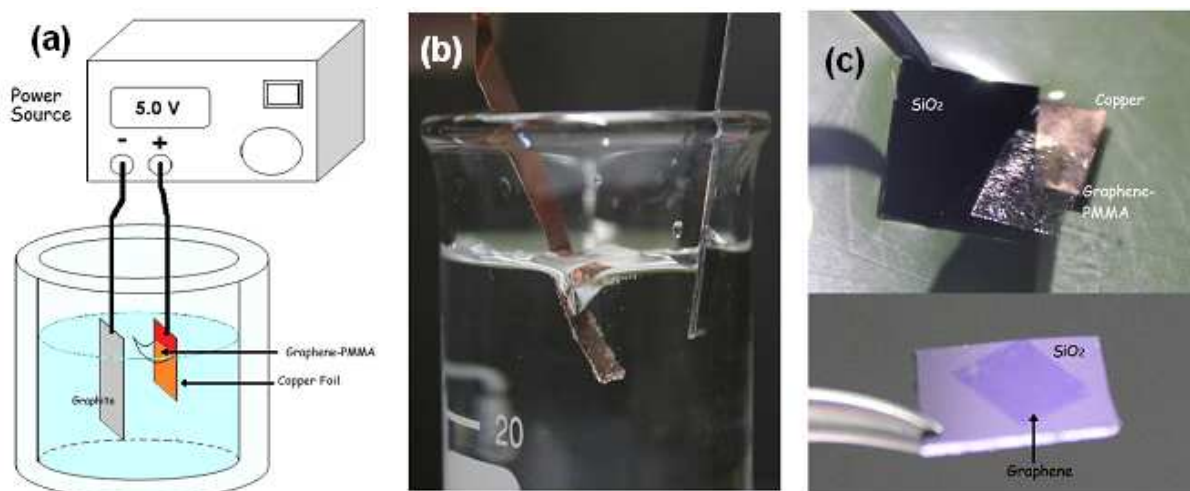


Figure 5. (a) System Diagram used for the electrochemical separation of graphene-polymer from the copper foil, (b) formation of hydrogen bubbles help to detach the graphene from the substrate, (c) graphene-PMMA stays on water and is transferred to a substrate SiO₂/Si.

6.3- GRAPHENE BY EXFOLIATION

i. Electrochemical Synthesis of graphene by exfoliation of HOPG

The electrochemical exfoliation was performed using a system conformed by a power source and electrochemical glass cell. Graphite plate was used as counter-electrode, HOPG as work electrode and a solution of sulfuric acid (5%) as electrolyte (Fig.6). For the exfoliation process was applied a potential of 10 V with pulses of 1 minutes.

This method yields flakes of few-layer graphene, which are detached from HOPG electrode when potential pulse is applied and they float over the surface of electrolyte due to hydrophobicity of graphene. The flakes were collected from solution surface using glass spatula and wash with deionized water and centrifuged a few times for eliminated of acid.

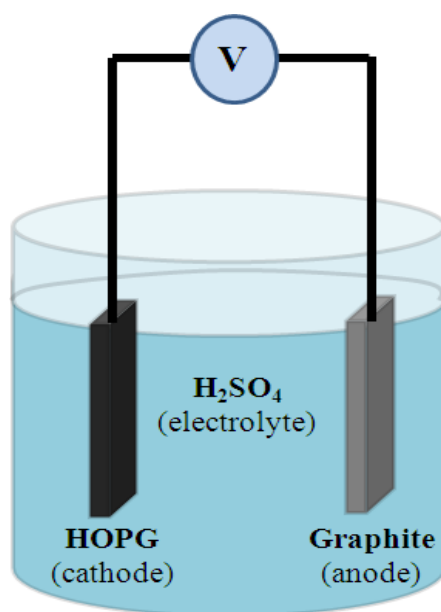


Figure 6. System Diagram shows electrochemical exfoliation of graphene from HOPG.

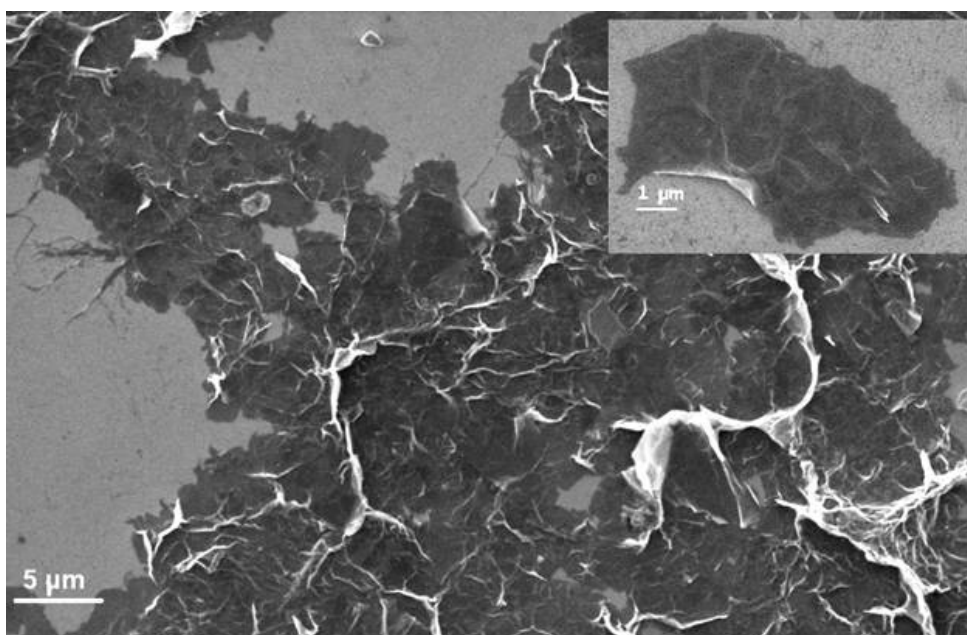


Figure 7. SEM image show multilayer graphene flakes obtained by electrochemical exfoliation of HOPG in aqueous sulfuric acid.

The flakes obtained were formed mainly by few and multi-layer graphene and was not found single-layer graphene (Fig.7). This method produces functionalized layers due what the potential applied is high and produced damage in the material as structural defects that can be functionalized by carbonyl and hydroxyl groups from the acidic environment of the electrolyte [16].

ii. Synthesis of graphene by chemical exfoliation of natural graphite

We used chemical synthesis of graphene by Hummer Modified method. Graphite natural was used as source of graphene. The graphite is put in a solution, compound of sulfuric and nitric acid (concentrate) for one hour in a ultrasonic bath, then, the graphite flakes was collected using centrifugation by eliminating the supernatant and wash them with deionized water and centrifuge again, this procedure is repeated many times for eliminating acid excess.

The graphite flakes were oxidized using a solution of K_2MnO_4 (5%) at $60^\circ C$ for 8 hours, which were put in a ultrasonic bath for 2 hours, this way, the flakes are exfoliated and formed a suspension of graphene oxide. Then, flakes were collected by centrifugation, eliminated the supernatant and washed with deionized; this procedure is repeated many times

Finally, using a hydrazine solution (1%), the graphene oxide flakes concentrated were reduced for 2 hours at 120°C. The flakes were concentrated by centrifugation and supernatant was eliminated, and washed using deionized water, this procedure was repeated many times

Material obtained consist in flakes of reduce graphene oxide. However, the reduction procedure was not efficient and large amount of graphene oxide was not reduced. Therefore, we obtained oxide and reduced-oxide graphene. The presence of oxide makes it difficult obtains images using SEM, since the material is less conductive in comparison to graphene.

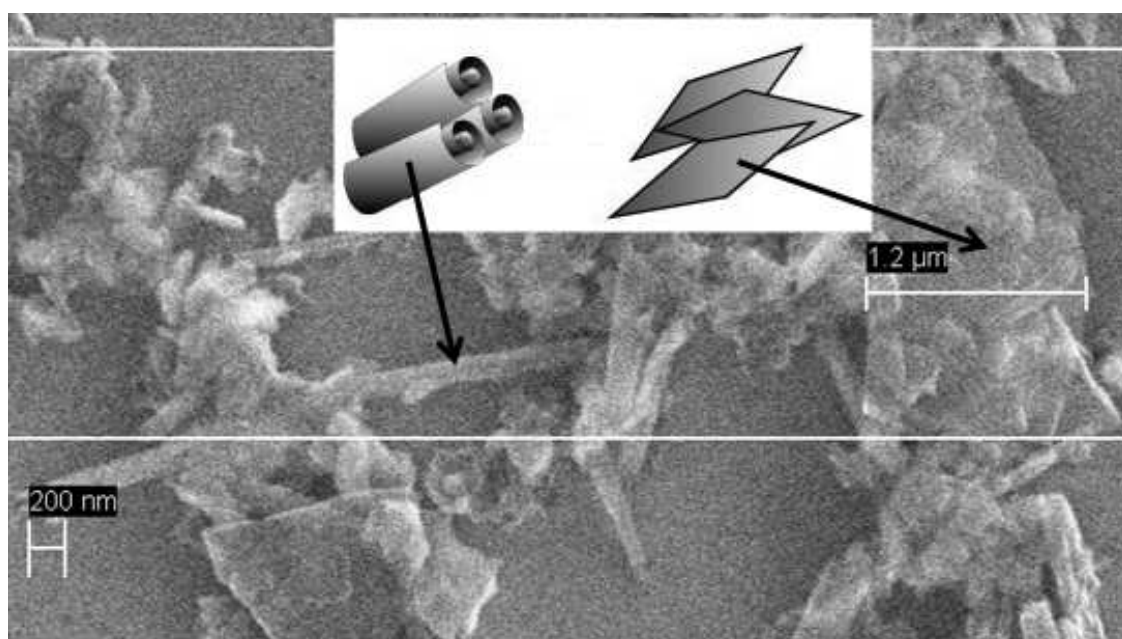


Figure 8. SEM image shows the rolls and flakes of reduced graphene oxide, obtained by exfoliation of natural graphite by the modified method Hummer.

In Fig.8, is possible to see the leaves reduced graphene oxide obtained. You can appreciate the presence of stretched and rolled sheets along with graphite residues. RGO flakes consist of several layers and the size varies from small sheets that appear rolled to large sheets that appear stretched.

The Fig.9 shows the Raman spectrum of RGO, it is possible appreciate that material consists of multilayer RGO, considering the shape of 2D band and the intensities ratio of 2 for bands G/2D.

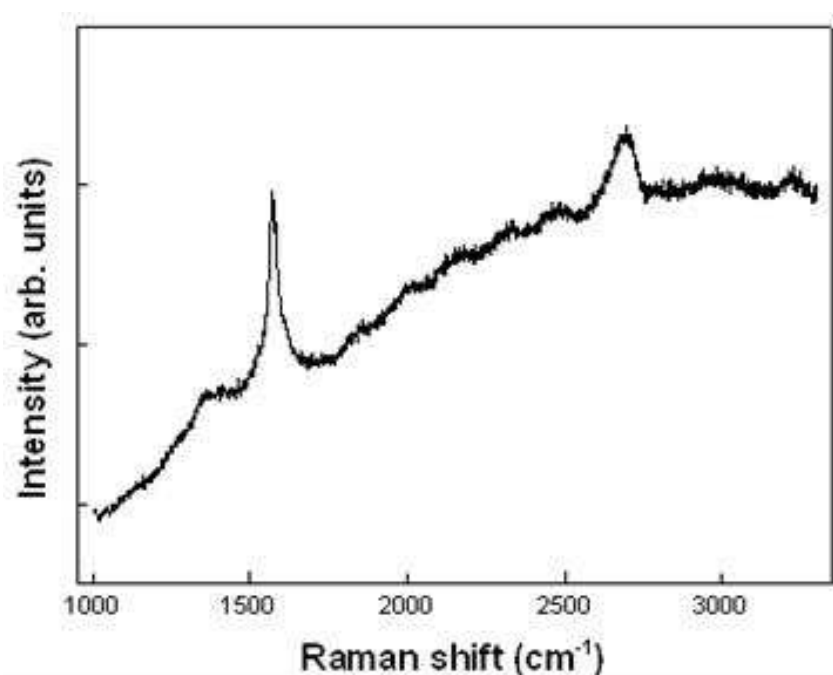


Figure 9. Raman spectrum flakes reduced graphene oxide obtained by chemical exfoliation using Hummer method ($\lambda = 532 \text{ nm}$)

7.- CONCLUSION

The graphene synthesis using various methods was possible. CVD synthesis shows the best results in terms of reproducibility, quality and size of graphene, being able to prepare material over large areas, from the optimal parameters established in this study. This material could be used as a protective film against corrosion, etc.

On the other hand, using exfoliation methods, both chemical and electrochemical, it is obtained as a result multilayer flakes. In the case of the chemical process, oxidized flakes were obtained mainly, despite reduction process that had carried out. This material could be used in applications for photovoltaic devices.

We are currently in the writing of manuscripts relating to the results of this research to be published in scientific journals.

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PUBLICATIONS

Title: CVD synthesis of graphene from acetylene catalyzed by a reduced CuO thin film deposited on SiO₂ substrates.

Authors: Andrea Cortés, Carlos Celedón and Ramón Zárate

Journal: Journal Of Chilean Chemical Society

Status: under review, send October, 2014.

Partially supported by AFOSR Grant FA9550-12-1-0060 and FONDECYT 11110522 (CHILE)

Abstract: Few-layer graphene was grown by Chemical Vapor Deposition on a CuO thin film pre-deposited by sputtering on SiO₂/Si substrates using acetylene as the carbon source. After evaporation of metal, graphene lies directly in contact with the SiO₂ dielectric layer. Raman spectroscopy was used to confirm the presence of a single and/or few-layers of graphene. This procedure does not requiring any post processing to transfer the thin film onto a dielectric substrate or the use of ultra-high vacuum during synthesis.

CONFERENCE PRESENTATION

Talk: Celdas fotovoltaicas y su modificación con nanoestructuras basadas en carbono

(Photovoltaic cells and its modification with carbon-based nanostructures)

Speaker: Andrea Cortes

Conference: 1er Taller en conversión de energía solar y térmica, en el marco del proyecto Anillo ACT1204

Universidad Católica del Norte, Antofagasta, CHILE

9-10, Oct. 2014.

Poster: Growth of graphene from acetylene on copper by CVD method

Authors: Andrea Cortes, Carlos Celedon

Conference: ICANM2014: International Conference and Exhibition on Advanced and Nano Materials, Calgary, CANADA

11-13, Aug. 2014

Poster: CVD Synthesis of graphene from acetylene on copper foil

Authors: Andrea Cortes, Carlos Celedon

Conference: Graphene 2014, Toulouse, FRANCIA

6-9, May. 2014

1.

1. Report Type

Final Report

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56-32-2654628

Organization / Institution name

Universidad Técnica Federico Santa María

Grant/Contract Title

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Graphene synthesis and characterization

Grant/Contract Number

AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".

FA9550-13-1-0060

Principal Investigator Name

The full name of the principal investigator on the grant or contract.

Andrea Cortes

Program Manager

The AFOSR Program Manager currently assigned to the award

Brett Pokines

Reporting Period Start Date

03/01/2014

Reporting Period End Date

03/01/2015

Abstract

Here is reported graphene synthesis by two methods, Chemical Vapor Deposition (CVD) using Acetylene as a Carbon source onto copper substrates, and the exfoliation of graphite (chemical and electrochemical). This methods have a great importance for applications in devices, because can be properly scaled to industrial level. Was studied the experimental parameters involved in the growth of graphene.

By Raman spectroscopy was made the characterization of material synthesized. CVD method allowed to grow single- bi- and few- layer graphene on copper. On the other hand, the exfoliation method produced multi-layer graphene flakes.

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Archival Publications (published) during reporting period:

none

Changes in research objectives (if any):

none

Change in AFOSR Program Manager, if any:

none

Extensions granted or milestones slipped, if any:

Due to delay in payment of the prize, the period of investigation was performed from Mar, 2014 to Mar, 2015.

AFOSR LRIR Number

LRIR Title

Reporting Period

Laboratory Task Manager

Program Officer

Research Objectives

Technical Summary

Funding Summary by Cost Category (by FY, \$K)

	Starting FY	FY+1	FY+2
Salary			
Equipment/Facilities			
Supplies			
Total			

Report Document

Report Document - Text Analysis

Report Document - Text Analysis

Appendix Documents

2. Thank You

E-mail user

Apr 03, 2015 23:22:29 Success: Email Sent to: andrea.cortes@usm.cl